



National Institute of Standards and Technology

Certificate of Analysis

Standard Reference Material[®] 154c

Titanium Dioxide

This Standard Reference Material (SRM) is intended primarily for use in the evaluation of techniques employed in the assay of titanium dioxide in the paint and ceramic industries. A unit of SRM 154c consists of a clear glass bottle containing 90 g of titanium dioxide (TiO₂) as a fine powder (< 45 μm).

Certified Value: The certified value, expressed as the mass fraction [1] of titanium dioxide (TiO₂) on a dry basis (see Drying Instructions), is provided below. A NIST certified value is a value for which NIST has the highest confidence in its accuracy in that all known or suspected sources of bias have been investigated or accounted for by NIST.

Titanium Dioxide (mass fraction): 99.591 % ± 0.062 %

This value is based on the results of 12 independent coulometric reductimetric back-titrations [2] performed on a stratified random sampled set of 8 units of SRM 154c and referenced to coulometrically standardized potassium dichromate (K₂Cr₂O₇). The dissolution and reduction procedure were adapted from American Society for Testing and Materials (ASTM) Standard Method D1394-76 [3]. The certified value includes corrections for elements known to interfere in the reduction and coulometric titration used for the certification. The uncertainty in the certified value is expressed as an expanded uncertainty, $U = ku_c$, calculated according to the methods in the ISO Guide [4]. The quantity u_c represents, at the level of one standard deviation, the potential combined effects of the uncertainty due to sample dissolution and reduction, coulometric titration, and correction for impurities. The quantity k is a coverage factor used to obtain an expanded uncertainty with an approximate confidence level of 95 %. The value of the coverage factor, $k = 2.004$, is determined from the Student's t -distribution with 55.05 degrees of freedom and a nominal confidence level of 95 %. The certified value is based on mass in vacuum. Corrections for the effect of air buoyancy were calculated using a density of 4.28 g/cm³ for SRM 154c. For use of SRM 154c as a calibration or evaluation standard, **it is the responsibility of the user to ascertain which elements interfere with the procedure being calibrated or evaluated and to make corresponding corrections for the given application.**

Expiration of Certification: The certification of SRM 154c is valid, within the measurement uncertainties specified, until **01 March 2011** provided the SRM is handled in accordance with the instructions given in this certificate (see Instructions for Use). This certification is nullified if the SRM is damaged, contaminated, or otherwise modified.

Maintenance of SRM Certification: NIST will monitor representative samples of this SRM over the period of its certification. If substantive changes occur that affect the certification before the expiration of this certificate, NIST will notify the purchaser. Return of the attached registration card will facilitate notification.

Coulometric analyses were performed by K.W. Pratt of the NIST Analytical Chemistry Division.

Statistical consultation was provided by W.F. Guthrie of the NIST Statistical Engineering Division.

The support aspects involved in the preparation, certification, and issuance of this SRM were coordinated through the NIST Standard Reference Materials Program by B.S. MacDonald.

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Certificate Issue Date: 01 June 2001

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INSTRUCTIONS FOR USE

Sampling: The unit should be thoroughly mixed by rotating the bottle before sampling. A minimum sample mass of 200 mg should be used for analytical determinations to be related to the certified value provided. The SRM should be stored, as received, in its original container with the cap tightly closed, under normal laboratory conditions.

Drying: A portion of SRM 154c should be dried for 2 hours at 110 °C prior to weighing the sample and stored in a desiccator over anhydrous magnesium perchlorate or equivalent desiccant, until analyzed.

SUPPLEMENTAL INFORMATION

Source of Material: The titanium dioxide used for this SRM was donated by CPM Industries, Wilmington, DE.¹ The material has the characteristic off-white color of rutile and was stated by the supplier as an Ultra High Purity grade of rutile titanium dioxide with a nominal crystal structure of > 99 % rutile.

Information Values: Noncertified impurity information values for SRM 154c are reported in Table 1. These values are based on the results of the determination of impurity trace elements in SRM 154c by glow-discharge mass spectrometry (GDMS). The values are given to provide additional characterization of the material only. They are not recommended for use to monitor or assess analytical performance.

Table 1. Noncertified Impurity Information Values

Element	mg/kg	Element	mg/kg	Element	mg/kg	Element	mg/kg
Aluminum	130	Europium	<0.05	Molybdenum	5.2	Silver	29
Antimony	3	Fluorine	<0.1	Neodymium	<0.05	Sodium	220
Arsenic	0.7	Gadolinium	<0.05	Nickel	0.2	Strontium	<3000
Barium	0.8	Gallium	0.7	Niobium	120	Sulfur	28
Beryllium	<0.01	Germanium	<0.1	Osmium	<0.05	Tellurium	<0.1
Bismuth	<0.05	Gold	<0.5	Palladium	<0.1	Terbium	<0.05
Boron	1.7	Hafnium	5	Phosphorus	100	Thallium	<0.05
Bromine	<0.1	Holmium	<0.05	Platinum	<0.1	Thorium	<0.01
Cadmium	<0.1	Iodine	<0.1	Potassium	31	Thulium	<0.05
Calcium	75	Iridium	<0.05	Praseodymium	<0.05	Tin	4.5
Cerium	<0.05	Iron	100	Rhenium	<0.05	Tungsten	1.6
Cesium	<0.5	Lanthanum	0.3	Rhodium	<0.5	Uranium	<0.01
Chlorine	11	Lead	1.1	Rubidium	5	Vanadium	7.8
Chromium	13	Lithium	0.9	Ruthenium	<0.05	Ytterbium	<0.05
Cobalt	0.07	Lutetium	<0.05	Samarium	<0.05	Yttrium	<200
Copper	14	Magnesium	17	Scandium	<0.05	Zinc	1.5
Dysprosium	<0.05	Manganese	1.5	Selenium	<0.1	Zirconium	190
Erbium	<0.05	Mercury	<0.1	Silicon	500		

¹Certain commercial organizations, services, equipment, or materials are identified in this certificate to specify adequately the experimental procedure. Such identification does not imply recommendation or endorsement by National Institute of Standards and Technology nor does it imply that the organizations, services, materials, or equipment identified are necessarily the best available for the purpose.

REFERENCES

- [1] Taylor, B.N., "Guide for the Use of the International System of Units (SI)," NIST Special Publication 811, 1995 Ed., (April 1995).
- [2] Pratt, K.W., *Anal. Chim. Acta*, **289**, p. 125, (1994) and *Anal. Chim. Acta*, **289**, p. 135, (1994).
- [3] ASTM D 1394-76 (reapproved 1999), "Standard Test Methods for Chemical Analysis of White Titanium Pigments," ASTM Book of Standards, **06.03**, West Conshohocken, PA.
- [4] *Guide to the Expression of Uncertainty in Measurement*, ISBN 92-67-10188-9, 1st Ed., ISO, Geneva, Switzerland, (1993); see also Taylor, B.N. and Kuyatt, C.E., "Guidelines for Evaluating and Expressing the Uncertainty of NIST Measurement Results," NIST Technical Note 1297, U.S. Government Printing Office, Washington, DC, (1994); available at <http://physics.nist.gov/Pubs/>.

Users of this SRM should ensure that the certificate in their possession is current. This can be accomplished by contacting the SRM Program at: telephone (301) 975-6776; fax (301) 926-4751; e-mail srminfo@nist.gov; or via the Internet <http://www.nist.gov/srm>.